such as the SPE disc, and the proliferation of automated techniques for performing the extractions, ensure that SPE will continue to be a preferred technique for sample preparation in many different analytical disciplines.

See also: **II/Extraction:** Solid-Phase Extraction. **III/Solid-Phase Extraction with Discs**.

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SOLID-PHASE EXTRACTION WITH DISCS

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Introduction

Solid-phase extraction is a well-established technique for the isolation, concentration and matrix simplification of analytes in samples with unfavorable properties for direct analysis by the best available approach. Extraction is achieved using a particulate sorbent packed into columns of short length (sometimes called 'cartridges') or immobilized in the form of a thin disc, referred to generically as 'disc technology'. Since the same sorbent chemistry is used for the extraction step and liquid desorption for the elution step in both approaches, the two techniques differ only in format. On an evolutionary scale, solid-phase extraction using short columns was introduced as a laboratory-scale technique in the late 1970s and came to prominence in the 1980s. Disc technology, by comparison, was first introduced in 1989, and is still evolving as a competitive technique to short packed columns. Simply stated, disc technology should be viewed as an alternative approach to performing solid-phase extraction with additional benefits and capabilities derived from the difference in format. Whereas packed columns are easily prepared in the laboratory for evaluating new sorbent chemistries and evaluating sampling properties, discs, so far, have only been produced in a manufacturing setting. Consequently, sorbent selection and device optimization have been restricted by market-driven considerations. As a consequence, the main applications of disc technology are generally narrowly focused on the needs of large volume users more so than is the case for conventional short packed columns.

Disc Formats

Solid-phase extraction discs are available in different styles and sizes. Particle-loaded membranes (Empore™ discs) contain 8–12 μm sorbent particles homogeneously distributed in a web of short poly (tetrafluoroethylene) (PTFE) fibrils. These are formed into 0.5-mm thick discs with diameters from 4 to 96 mm. They are flexible and superficially resemble filter paper discs. They are used with some supporting structure such as a frited glass filter or porous plastic support. The discs contain about 90% by weight of sorbent with the balance being the PTFE microfibrils. Some characteristic physical properties are indicated in **Table 1**. Particle-loaded membranes are also available in a syringe barrel format similar to conventional short packed column-sampling devices. In this case, the sorbent bed contains particles of a larger diameter, about 50 μm, in thicker discs, about 1.0 mm, sealed into the base of a 4 mm (1 mL), 7 mm (3 mL), 10 mm (6 mL) and 20 mm diameter (40 mL) open syringe barrel. These discs have an integral prefilter consisting of a graded density of poly(propylene)

Table 1 Rough guide to the physical properties of solid-phase extraction disc according to disc diameter

Property	*4 mm*	*7 mm*	*10 mm*	*25 mm*	*47 mm*	*90 mm*
Surface area (cm^2)	0.13	0.38	0.80	4.9	17	64
Bed mass (mg)[a]	4	10	25	140	500	1850
Flow rate (mL min^{-1})	0.5	1.5	3	20	60	250
Elution volume (mL)	0.15	0.25	0.5	3	10	35
Typical sample volume (mL)	< 1	< 5	< 25	< 250	< 1000	< 5000

[a]Silica-based sorbents.

microfibres on the top sampling surface. They are recommended as a replacement for short packed column-sampling devices as well as for processing small volumes of viscous biological fluids. Particle-embedded glass fibre discs (SPEC™ discs) contain particles of a narrow size distribution, about 10–30-μm diameter, woven into a glass fibre-supporting matrix. The smaller diameter discs are rigid and self-supporting but large-diameter discs require a supporting structure similar to the particle-loaded membranes. Particle-embedded glass fibre discs are also available with a depth filter region of 0.1–0.2 mm combined with a sorbent extraction region of 0.8–0.9-mm thickness. Laminar discs (Speedisks™) contain 10 μm sorbent particles in a consolidated 0.5- or 1-mm thick bed (usually) retained by two glass-fibre filters held in place by screens and a retaining ring in a preassembled cartridge with a 50-mm diameter sampling area. This configuration is designed to provide high sample flow rates for extracting large volumes of water.

Solid-phase extraction discs are available as loose discs for use in filtration-style apparatus for large volume samples and in open syringe barrels and cartridges for extracting intermediate and small sample volumes. These forms are compatible with sample processing using suction, positive pressure, syringe filtration and centrifugation. Common sorbents include octadecylsiloxane- and octylsiloxane-bonded silica particles, styrene-divinylbenzene porous polymers, porous polymer and silica-based cation and anion exchangers, mixed mode sorbents, activated carbon and immobilized crown ether sorbents. Other sorbents could easily be produced in a disc format if a sufficient market to support their production could be identified. Solid-phase extraction discs have been used primarily for sample preparation prior to chromatographic analysis. The disc format supports other, if minor applications at present, such as *in situ* detection using radioactivity counting, phosphorescence, and matrix-induced laser desorption mass spectrometry, etc. SPEC™ discs can be inserted directly into a hole in a glass fibre thin-layer sheet and developed in a conventional manner combining recovery and separation into a single step. This approach is commonly used for toxicological screening of biological fluids.

Advantages of Disc Technology

The change in format from a short packed column to a disc has some attendant advantages for solid-phase extraction. These can be briefly summarized as follows. The larger cross-sectional area of discs and decreased pressure drop compared to conventional column-like sampling devices results in shorter sample processing times and decreased plugging by suspended particle matter. This is important in environmental surveillance programmes, such as the analysis of surface waters for persistent or toxic substances, where large sample sizes are common to obtain adequate detection limits and samples are often burdened by suspended particle matter. The large surface area per unit bed mass of the discs facilitates passive sampling approaches to solid-phase extraction and related uses as an indirect monitor of bioconcentration (discussed later).

The use of smaller diameter sorbent particles and the greater stability of the sorbent bed results in improved kinetic performance and reduced channelling. This allows the use of a smaller bed mass for extraction and results in less variation between sampling devices. The reduced bed mass provides cleaner sample backgrounds and lower interferences by minimizing nonspecific matrix adsorption. Smaller bed masses allow miniaturization of sampling devices for convenient handling of small sample sizes together with smaller elution volumes for analyte recovery. They also facilitate novel sampling approaches such as in-vial elution and on-disc derivatization (discussed later).

Kinetic Characteristics

Forced-flow planar chromatography has been used to study the kinetic properties of octadecylsiloxane-bonded silica particle-loaded membranes and particle-embedded glass fibre discs (**Table 2**). The total porosity of the discs is about 0.50, comprised mainly of interparticle porosity (about 0.40) with a large

Table 2 Kinetic properties of octadecylsiloxane-bonded silica solid-phase extraction discs

Property	*Particle-loaded membranes*	*Particle-embedded glass fibre discs*
Total porosity	0.52–0.54	0.51
Interparticle porosity	0.37–0.48	0.47
Intraparticle porosity	0.06–0.15	0.04
Specific permeability (10^{-14} m^{-2})	2.2–2.5	8.4
Flow resistance parameter	1000–1250	900–1000
Apparent particle size (μm)	5.8–7.7	15.3
Nominal pore diameter (nm)	6	8
Minimum plate height (μm)	56	
Optimum mobile phase velocity (mm s^{-1})	0.13	
Coefficients for Knox equation		
A	3.75 (0.5–1.5 for columns)	
B	1.72 (1.0–4.0 for columns)	
C	1.54 (0.05–0.7 for columns)	

fraction of the particle pore volume inaccessible to the mobile phase. This is not unusual for porous silica sorbents with a high loading of bonded phase restricting access to the pore volume. It is also compatible with the desire for a high sorption capacity and is not necessarily an undesirable feature. The specific permeability and flow resistance parameter support the hypothesis that the disc structure is homogeneous and devoid of through pores (holes), an essential requirement for a thin sampling medium. The apparent particle size for the particle-loaded membranes, about 7 μm, and particle-embedded glass fibre discs, about 15 μm, are in reasonable agreement with the manufacturers' claims given the assumptions used in calculations for converting the pressure–flow relationships to particle size values. The particle-loaded membrane provides an optimum plate height of 56 μm at a linear velocity of 0.13 mm s^{-1}. A 0.5-mm disc of 47-mm diameter will provide between 4 and 9 theoretical plates over the flow rate range of 5–100 mL min^{-1} with a maximum value at 13 mL min^{-1}. Compared to typical slurry-packed columns, the contribution of both flow anisotropy and resistance to mass transfer to the plate height are unusually large for the particle-loaded membranes, and while the bed may have an homogeneous structure, it does not have an ideal kinetic structure. Fortunately, large plate numbers are not required for efficient extraction.

Disc Selection

The parameters of interest in selecting a disc for a particular application are size, sorbent chemistry and sample capacity. Referring to Table 2, large-diameter discs are used for processing large sample volumes to improve sample throughput. Small-diameter discs are used for processing small sample volumes and to recover analytes in a small solvent volume to eliminate the need for solvent evaporation prior to analysis. Small-diameter discs are frequently used in clinical, forensic and pharmaceutical analysis and large-diameter discs in environmental analysis. The same sorbents used for conventional solid-phase extraction are generally used for disc extraction, but since discs are used for a narrower range of applications at present, the number of frequently used sorbent types is smaller (**Table 3**). The majority of applications proposed to date involve sampling of aqueous solutions. Octadecylsiloxane-bonded silica, poly(styrene-divinylbenzene) and activated carbon sorbents are used for general reversed-phase sampling; porous polymer and silica-based cation and anion exchangers are used for isolating ionizable and ionic compounds; and cheltaing ligands for the selective isolation of metals (particularly precious metals and radionuclides). Discs with different sorbent chemistries can be stacked on top of each other and analytes recovered in a single elution or as groups after physically separating the discs. Stacked discs of the same or varied sorbent chemistry are useful for extracting complex samples containing compounds that differ significantly in polarity or ionization, for achieving larger breakthrough volumes, and as an approach for reducing interferences by the selective sorption of the matrix by one disc and the analytes by the other. Stacked discs of a reversed-phase and cation exchange type are a suitable alternative to mixed-mode sorbents for isolating drugs and their metabolites from biological fluids. Discs containing porous polymer sorbents have been proposed for air sampling, particularly as a replacement for poly(urethane) foams, but have not been widely adopted.

In recent years, disc-based solid-phase extraction has become a popular technique for sample cleanup in ion chromatography and capillary electrophoresis. The interfering ions, often at relatively high concentrations, can mask, broaden or change the migration time of the ions of interest. The Novo-Clean™ discs

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Table 3 Sorbent selection for disc applications

Sorbent	*General applications*
Octadecylsiloxane- and octylsiloxane-bonded silica	Octadecylsiloxane-bonded sorbents are widely used for reversed-phase sampling by non-specific sorption (and are more widely used than octylsiloxane-bonded sorbents). There are many applications to the extraction of non-polar and moderately polar pesticides (all classes), herbicides, phthalate and adipate esters, polycyclic aromatic compounds, food additives, pharmaceutical compounds, hydrocarbons and grease, etc. from aqueous solution. A large number of approved methods for water analysis.
Poly(styrene-divinylbenzene)	Used for compounds poorly extracted by octadecylsiloxane-bonded silica sorbents because of high water solubility such as polar pesticides, herbicides, phenols and pharmaceutical compounds. Lightly sulfonated, acylated and hydroxymethylated polymers claimed to provide higher recovery of neutral polar compounds because of better surface compatibility with aqueous samples.
Activated carbon	Applications similar to poly(styrene-divinylbenzene) but less frequently used. Methods proposed for triazine herbicides, some polar pesticides and *N*-nitrosodialkylamines.
Mixed mode	Usaully co-bonded octadecylsiloxane and benzenesulfonic acid groups (or sorbent mixtures) used predominantly in clinical toxicology and pharamaceutical analysis for the simultaneous isolation of drugs and their metabolites. A number of established methods for drugs of abuse (marijuana and cocaine metabolites, amphetamines, phencyclidine and opiates) in biological fluids.
Sulphonic acid and quaternary amine ion exchangers	Selective isolation of ionic and easily ionizable compounds. Many methods for acidic herbicides and pesticides in water and basic drugs in biological fluids. Porous polymer sorbents are stable over the whole pH range. Sulphonated cation exchange polymers in the hydrogen, silver or barium forms used for cleanup of samples analysed by ion chromatography and capillary electrophoresis.
Crown ethers	Silica or other support with tethered mixed oxygen–nitrogen donor cryptands used for the selective isolation of metals by molecular recognition mechanisms. Commonly used for the isolation of precious metals (Pd, Pt, Rh) and radionuclides (Cs, Sr, Pb) from high concentrations of other metals to determine environmental burden and for dating geochemistry samples. Other applications include the removal of base metal impurities (Bi, Sb, Fe, Pb, Bi, Cu, Hg) from refinery streams, plating baths, etc.

contain a sulfonic acid-functionalized resin in the hydrogen, silver or barium forms housed in a cartridge adapted for syringe filtration. The hydrogen form is used to remove hydroxide and carbonate ions from samples by neutralization. The silver form is used to remove excess halide ions from samples by formation of insoluble silver halide salts that precipitate in the disc matrix. **Figure 1** shows an example of the detection of nitrate, fluoride and phosphate in a hydrochloric acid digest of a paper coating by capillary electrophoresis. The barium form is used to remove sulfate through the formation of insoluble barium sulfate.

Sample Processing

A generic outline for processing aqueous samples using disc technology is presented in **Table 4**. This can be rescaled for different disc and sample sizes using the data in Table 1. The sampling process begins with decontamination of the disc by rinsing with organic solvent to remove impurities followed by solvent conditioning to facilitate effective and reproducible sorption of the analytes. Before the sample is added to the disc the conditioning solvent is rinsed from the disc with water to avoid premature breakthrough of analytes. For large sample volumes, a small amount of organic solvent (1–5% v/v) is added to aqueous samples to maintain a constant sample velocity. This usually has little influence on the breakthrough volumes except for porous polymer sorbents with a low degree of crosslinking. The selective adsorption of organic solvent by the polymer changes the sorbent volume and selectivity, resulting in changes in the breakthrough volume of up to an order of magnitude when either methanol,

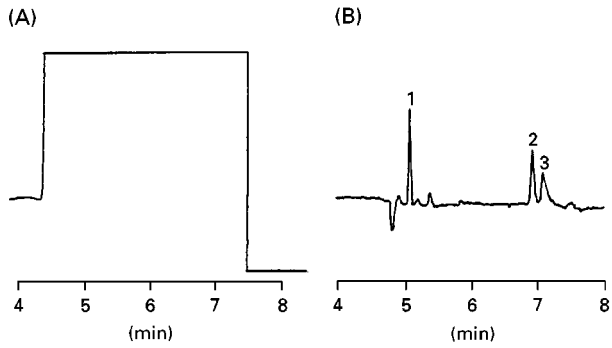

Figure 1 Separation of trace concentrations of nitrate (1), fluoride (2) and phosphate (3) anions (1–10 p.p.m.) from a hydrochloric acid digest of a paper coating without treatment (A) and after disc cleanup using a Novo-Clean IC-Ag disc (B) by capillary electrophoresis. (Reproduced with permission from Saari-Nordhaus R and Anderson JM (1995) Membrane-based solid-phase extraction as a sample clean-up technique for anion analysis by capillary electrophoresis. *Journal of Chromatography A* 706: 563–569. Copyright Elsevier Science.)

2-propanol, acetonitrile or tetrahydrofuran are used as sample processing solvents (**Table 5**). These results are not in any way artifactual and are adequately predicted by the solvation parameter model for the breakthrough volumes. A sample processing solvent is not usually required for small sample volumes. Viscous samples may be easier to handle if diluted with either water or an organic solvent to increase their sample-processing rate. Samples containing a significant amount of particulate matter should be either prefiltered, processed with a filter aid (cover the surface of the disc with 1 cm of 40-μm glass beads), processed by using a combination of a disc with an integral depth filter, or processed after adjusting the pH to dissolve soluble particles. Weak acids and bases are extracted by ion exchange or by ion suppression reversed-phase sampling at a suitable pH. Increasing the ionic strength of the sample (e.g. adding sodium chloride) may improve the extraction of neutral compounds in reversed-phase extractions. The drying stage is important because water retained by the disc (and the disc support structure) contaminates the elution solvent and may cause difficulty if the solvent is to be reduced in volume or the analytes analysed by gas chromatography. Water may also reduce the efficiency of the elution step, particularly for elution solvents only partially miscible with water. Discs are usually dried by suction, but other possibilities include freeze–drying or desiccation over a drying agent with or without vacuum. For silica-based sorbents, a masking agent such as triethylamine (1% v/v) is sometimes added to the eluting solvent to increase the recovery of basic compounds strongly retained by interaction with silanol groups. Supercritical fluid extraction with carbon dioxide has been used to recover analytes isolated by disc extraction from water. In this case, the discs were used for preconcentration and media exchange since supercritical fluid carbon dioxide provides a poor extraction medium for aqueous solutions.

Passive Sampling and Bioconcentration

Passive sampling involves the extraction of organic compounds by immersion of the sampling disc in aqueous samples as opposed to filtering samples through the extraction disc. This process is convenient but little explored. One reason is the slow equilibrium of the extraction process even for stirred solutions. The uptake of organic compounds in a stirred solution depends on the size of the disc, time and the degree of mixing. Uptake by the disc may never be complete in a reasonable time but greater than 80%

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Table 4 Generic guide for sample processing using solid-phase extraction discs. In this example, a 47-mm disc and a 1-litre water sample are used for illustration

Decontamination	With disc installed in filtration apparatus (or cartridge) rinse with 10 mL of solvent (acetonitrile) by allowing the solvent to soak into the membrane for a few minutes and then remove by suction.
Condition	As above but using 10 mL of methanol. Before the last drop of methanol has been sucked through the disc, add 10 mL of deionized water. The disc should not be allowed to suck dry until after the sample has been processed. It may be necessary to break the vacuum for ease of manipulating solutions.
Sample	The sample containing 1% (v/v) methanol is passed through the disc with a suitable reservoir in place. Filter aid or a prefilter may be required for samples with a heavy burden of particulate matter. The sample is processed at a flow rate between about 50 and 100 mL min^{-1} using a vacuum of about 10–20 mmHg.
Drying	Bulk water is removed from the disc and sampliing appratus by sucking air through the disc under full vacuum for about 3 min. Volatile compounds may be lost.
Recovery	The analytes are recovered by passing two 5-mL volumes of acetonitrile through the disc. The first volume of acetonitrile is allowed to soak into the disc for a few minutes and then gently sucked through the disc. Without letting the disc run dry, the second volume of solvent is added to the disc and sucked through the disc. The disc is then allowed to suck dry.

Table 5 Breakthrough volumes (cm^3) of some organic compounds on porous polymer particle-loaded membrane discs with 1% (v/v) organic solvent in water

Compound	*Acetonitrile*	*Tetrahydrofuran*	*2-Propanol*	*Methanol*
Anisole	575	300	1750	1300
Acetanilide	95	25	100	75
2-Phenylethanol	150	25	100	200
Heptan-1-ol	2700	300	2700	1000
Propyl propanoate	1400	200	1250	750

extraction of pesticides in an agitated 500 mL sample using a 47-mm diameter particle-loaded membrane in 24 h was reported. Octadecylsiloxane-bonded silica discs have been explored as surrogate models for bioconcentration. This process is referred to as 'biomimetic extraction' and is used to estimate the selective uptake of organic compounds by aquatic species. Biomimetic extraction results in the preferential concentration of the more hydrophobic compounds from water and is theorized to provide a more realistic approach to assessing environmental effects of soluble organic compounds than results obtained by total extraction techniques. Measurements are made using passive disc sampling under conditions that avoid sample depletion to emulate the sorption of organic compounds by aquatic species. This is a relatively new approach to toxicity assessment and requires further work to establish a satisfactory relationship between the sorption properties of the disc and those of target aquatic species.

In-Vial Elution and Derivatization

The in-vial elution method reduces the amount of organic solvent used for recovery and eliminates tedious sample-preparation steps compared to conventional methods. This technique is an equilibrium-based approach for the recovery of extracted analytes. After extraction, the dried disc is placed in an autosampler vial and covered with a few mL of solvent. Recovery depends on the selection of the solvent, temperature and equilibration time. An equilibration time of 4 h, or overnight, at room temperature is sufficient in many cases to provide acceptable recovery ($> 90\%$) and method precision as well as being compatible with automated sample analysis in gas and liquid chromatography. Addition of a derivatizing reagent to the solvent used for desorption allows both steps to be performed simultaneously in the same vial without removal of the disc. The vial can be sealed and heated to enhance the rate of derivatization. Common reactions employed so far include trimethylsilylation and alkylation for gas chromatographic analysis but other reagents should be equally applicable. It was demonstrated that ion exchange discs catalyse the rate of alkylation of acid herbicides, surfactants and pesticides using a solution of an alkyliodide as the derivatizing reagent. An example is shown in **Figure 2**.

Preservation and Storage

Samples or their extracts frequently have to be stored between sampling and analysis. Compounds differ in their stability to various storage conditions. The degradation or loss of pesticides and other organic compounds stored in water is mainly due to hydrolysis, biodegradation, photolysis and evaporation. Solid-phase extraction discs provide a convenient storage medium for compounds recovered from natural waters. General results indicate that discs provide equivalent or greater stability to the storage of pesticides in water at 4°C containing inhibitors to reduce

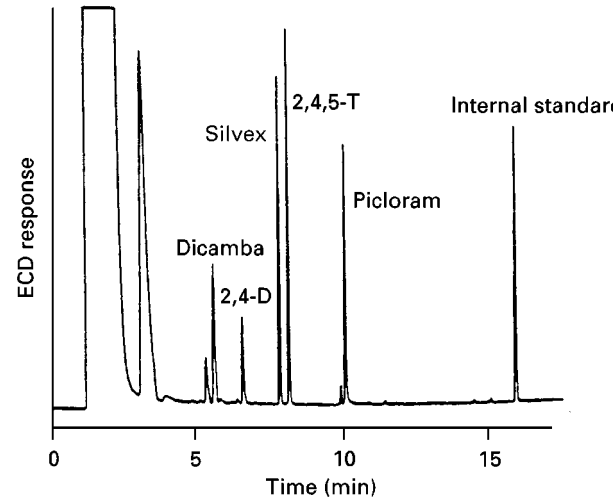

Figure 2 Use of in-vial elution and derivatization for the determination of chlorinated acid herbicides in an extract from a spiked ($< 10\ \mu g\ L^{-1}$ each) river water sample by gas chromatography. A 500-mL sample was processed through a 25-mm strong anion-exchange disc, the disc dried by suction, sealed in an autosampler vial with 1 mL of acetonitrile and 0.2 mL of methyl iodide and heated at 80°C for 1 h, and excess methyl iodide removed by evaporation. (Reproduced with permission from Field JA and Monohan K (1996) Chlorinated acid herbicides in water by strong anion-exchange disc extraction and in-vial elution and derivatization. *Journal of Chromatography A* 741: 85–90. Copyright Elsevier Science.)

biodegradation. One of the primary mechanisms of disc degradation was identified as hydrolysis resulting from contact with water remaining in the disc after drying by suction. Stability was increased by desiccation of the discs. Stability has to be judged on an individual compound basis but discs provide a more efficient and convenient medium for extract preservation. Their low weight and small size make them a useful medium for shipping.

Automation and Multi-Well Formats

The introduction of solid-phase extraction discs occurred at a time when the automation of common laboratory process was very much in vogue for improving the quality of laboratory information and to reduce operating costs. Most disc extraction processes can be automated. Series-coupled filtration devices are popular for parallel processing of environmental water samples. Disc sampling devices in cartridge format are compatible with the automated and robotic workstations in use for automated sample preparation using conventional packed column formats. Multi-well microtitre plates have been adapted to disc extraction by incorporating the disc in the bottom of the well and using automated liquid-handling systems for high throughput extractions. This is achieved by a combination of automated parallel processing and eluent collection without volume reduction in a matching multi-well plate compatible with standard autoinjection devices. This approach meets the high sample throughputs demanded of current applications in bioanalysis and combinatorial chemistry for drug discovery. Short precolumns containing several discs stacked in series or packed with a spiral of particle-loaded membrane have been used for online extraction combined with liquid chromatography for pesticide monitoring of drinking and surface water samples. This is not an area that extraction discs dominate, and although useful and successful, clear advantages over short packed columns have not been demonstrated.

Future Developments

Most developments in disc technology are recent enough that they are still to some extent regarded as novel rather than routine laboratory tools. They are expected to continue to replace conventional short packed columns in those applications where the disc format has specific advantages. In addition, disc technology has already defined new opportunities for expansion of solid-phase extraction in passive sampling, storage and preservation of extracts, field sampling, and as an indirect indicator of bioconcentration. Since discs are not laboratory-made products, the variety of disc chemistries available is linked to the identification of a viable market for a commercial product. For the present, this has tended to decouple efforts to promote disc technology from modern research on sorbent chemistry for the next generation of solid-phase extraction products.

See also: **II/Extraction:** Solid-Phase Extraction. **III/Sorbent Selection for Solid-Phase Extraction.**

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SOLID-PHASE EXTRACTION: SORBENT SELECTION

See **III / SORBENT SELECTION FOR SOLID-PHASE EXTRACTION**

SOLID-PHASE MATRIX DISPERSION: EXTRACTION

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Introduction

Matrix solid-phase dispersion (MSPD) is a patented analytical process for the preparation, extraction and fractionation of solid and/or viscous biological samples prior to instrumental or other forms of analysis. MSPD involves the direct mechanical blending of samples with standard solid-phase extraction (SPE) bonded-phase solid support materials. In this process, the bonded-phase support acts as both an abrasive to produce disruption of sample architecture and as a 'bound' solvent that assists in accomplishing sample disruption. The sample is dispersed over the surface of the bonded-phase support material, producing a unique mixed-character phase for conducting target analyte isolation. This process has been applied to the isolation of a wide range of drugs, pollutants and other compound classes from a variety of sample matrices. The factors affecting the use of MSPD and its applications for sample preparation, extraction and fractionation are addressed.

Development of MSPD

The classical application of all forms of liquid chromatography requires that the sample be applied in a liquid state to the head of the column. Thus, in order to accommodate the use of solid-phase extraction (SPE) columns and discs in the development of an analytical procedure, methodology must also be developed to render the sample and target analytes contained therein into a liquid, relatively non-viscous, particulate-free and homogeneous condition. While some biological fluids are readily obtained in this form, most biological samples do not start out being directly applicable to SPE. This presents the analyst with some rather unique opportunities to apply or develop the best process for rendering a sample into a form compatible with liquid chromatography. Indeed, the most difficult and complex samples to analyse are the solids and semi-solids that are derived from a biological origin. Such samples may be obtained from animal or vegetable material and consist of a non-homogeneous array of fat and/or other tissues, fibre, pulp, etc.

For these and other reasons, the preparation of biological samples for SPE requires an initial disruption of the gross architecture of the sample. This step in the process assures access to all of the components of a sample and initiates the necessary homogeneity required for analysis. Disruption and homogenization also provide a larger overall surface area, generating greater access to solvents and reagents used for analyte isolation, which is the next step in the process. Samples that are by their nature already reasonably homogeneous, such as milk, fruit juices, plasma, etc., are less complicated in this regard but may be too viscous or contain particulates that could hinder rapid SPE extraction and fractionation. For such samples, dilution, filtration and/or centrifugation often solve these problems.

Classical processes for solid or semi-solid sample disruption usually involve one or various combinations